भारतीय मानक Indian Standard

IS 1055: 2023

निकोटीन सल्फेट विलयन — विशिष्टि

(दूसरा पुनरीक्षण)

Nicotine Sulfate Solution — Specification

(Second Revision)

ICS 65.100.10

BIS 2023



भारतीय मानक ब्यूरो BUREAU OF INDIAN STANDARDS

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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Divisional Council.

Nicotine sulfate solution is extensively used in the control of insect pests of agricultural importance. It is manufactured from waste tobacco and from the liquors obtained from factories making chewing and smoking tobacco. The waste tobacco is macerated with water and lime, and then steam-distilled. The distillate is neutralized with sulphuric acid and concentrated.

Nicotine sulfate solution is manufactured to contain 40 percent (m/m) of nicotine.

This standard was first published in 1957. Subsequently one amendment was issued. In first revision standard was being revised to make its requirements up to date.

In year 2020 vide S.O. 1196 (E) dated 20 March 2020, the nicotine sulfate was banned by Government of India for domestic use but continued to manufacture for export. In this revision, the standard has been brought out in the latest style and format of the Indian Standards. It also incorporates one amendment issued to this standard.

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1988 and the Rules framed thereunder and *Standards of Weights and Measures (Packaged Commodities), Rules* 1977. However, this standard is subject to the restrictions imposed under the *Act* and Rules wherever applicable.

The composition of the Committee responsible for the formulation of this standard is listed in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed, or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

NICOTINE SULFATE SOLUTION — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for nicotine sulfate solution.

2 REFERENCES

The standards, given below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards.

IS No.	Title
IS 1070 : 1992	Reagent grade water (third revision)
IS 8190 (Part 2) : 1988	Requirement for packing of pesticides: Part 2 Liquid pesticides (second revision)
IS 10627 : 1983	Methods for sampling of pesticidal formulations

3 REQUIREMENTS

3.1 Description

The material shall be a clear, yellowish brown to deep, brown aqueous solution of the dibasic salt, nicotine sulfate, and may have acrid odour. It shall be free from sediment, scum, sludge, suspended matter or other extraneous impurities.

3.2 Nicotine Content

The material shall contain not less than 40.0 percent by mass of nicotine when tested by the method prescribed in Annex A.

3.3 Sedimentation

The material shall satisfy the requirement of the sedimentation test prescribed in Annex B.

4 PACKING

The material shall be packed as per requirements given in IS 8190 (Part 2).

5 MARKING

5.1 The containers shall be securely closed and shall

bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name and address of the manufacturer;
- c) Batch number;
- d) Date of manufacture;
- e) Date of expiry;
- f) Net quantity;
- g) Nominal simazine content, percent (m/m);
- h) Cautionary notice as worded in the *Insecticides Act*, 1968, and Rules framed thereunder; and
- j) Any other information required under the Legal Metrology (Packaged Commodities) Rules, 2011.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 within 90 days of its manufacture. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627. However, the criteria for conformity of the material when tested, shall he the limits of tolerances, as applicable over the declared nominal value and given under clause 3.2 of this standard.

7 TESTS

7.1 Tests shall be carried out by the appropriate methods referred to in clause 3.2 and 3.3.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A (Clause 2.2)

DETERMINATION OF NICOTINE CONTENT

A-1 METHOD

A-1.1

The method consists in steam distilling the nicotine from an alkaline medium, converting it into a salt by collecting the distillate in an acid medium, precipitating with silicotungstic acid and finally weighing the precipitate.

A-2 REAGENTS

A-2.1 Liquid Paraffin

A-2.2 Sodium Hydroxide Solution — Aqueous,

40 percent (m/v).

A-2.3 Phenolphthalein Indicator Solution — 1.0 percent (m/v). Dissolve 1 g of phenolphthalein in 60 ml of rectified spirit and dilute to 100 ml with water.

A-2.4 Dilute Hydrochloric Acid — (1:4) and $(1:100\ 0)$ by volume.

A-2.5 Silicotungstic Acid Solution — Dissolve 120 g of silicotungstic acid (4H₂O.SiO₂. 12WO₃.22H₂O) in water and dilute to one litre. The solution should be free from cloudiness and green in colour.

 $\label{eq:NOTE} \begin{array}{llll} \mbox{NOTE} & -- \mbox{The silicotungstic acid should be white or pale} \\ \mbox{yellow crystals free from green colour. Of the several} \\ \mbox{silicotungstic acids, } & 4H_2O.SiO_2.10WO_3.3H_2O & \mbox{and} \\ \mbox{4H}_2O.SiO_2.12WO_3.20H_2O & \mbox{do not give crystalline} \\ \mbox{precipitates with nicotine and should not be used.} \end{array}$

A-2.6 Methyl Orange Indicator Solution — 0.04 percent (m/v), in aqueous ethyl alcohol (20 percent by volume).

A-3 PROCEDURE

A-3.1 Weigh accurately 0.5 g to 1.0 g of the material in a suitable tared weighing tube. Quantitatively transfer the weighed material to a 500 ml Kjeldahl flask using water to wash the last traces of the material into the flask. If necessary, add to the contents of the Kjeldahl flask a little of liquid paraffin to prevent frothing during distillation, add a few small pieces of pumice to prevent bumping. Make the contents of the flask alkaline by adding a slight excess of sodium hydroxide solution, using phenolphthalein as indicator. Fit the mouth of the Kjeldahl flask with a two-holed rubber stopper through which passes the stem of a trap bulb and an inlet tube for steam. Connect the free end of the trap bulb to a well cooled condenser, the lower end of which dips below the surface of 10 ml of dilute hydrochloric acid (1:4) contained in a suitable receiving flask.

Connect the inlet tube to the source of steam and distil rapidly with the current of steam. When the distillation is well under way, heat the Kieldahl flask using a Bunsen burner to reduce the volume of the contents of the flask as far as practicable without causing bumping or undue separation of insoluble matter. Continue the distillation until a small quantity of the distillate shows no cloudiness or opalescence when treated with a drop of silicotungstic acid solution and a drop of dilute hydrochloric acid (1:4). Confirm the alkalinity of the residue in the Kjeldahl flask phenolphthalein indicator solution. Reduce the volume of the distillate by concentrating it on a steam bath (see Note) and make up the volume of the concentrating distillate to a convenient volume in a volumetric flask with water at room temperature. Thoroughly mix the contents of the volumetric flask and filter through a dry filter paper, if it is not clear. Collect the filtrate in a convenient flask. Test a portion of this filtrate with methyl orange indicator solution to confirm its acidity.

NOTE — By heating on a steam bath the nicotine content of the distillate is not affected.

A-3.2 Pipette an aliquot (see Note) of the filtrate, containing about 0.1 g of nicotine, into a beaker and add at the rate of 3 ml of dilute hydrochloric acid (1:4) for each 100 ml of the aliquot and one millilitre of silicotungstic acid solution for every 0.01 g of nicotine supposed to be present in the aliquot. Stir the contents of the beaker thoroughly and let stand overnight at room temperature. Before filtering, stir the contents of the beaker to see that the precipitate settles down quickly and is in a crystalline form. Filter the contents of the beaker through an ashless filter paper. Wash all the residue remaining in the beaker on to the filter using dilute hydrochloric acid (1:1000) at room temperature. Wash the filter with dilute hydrochloric acid (1:1000) until a few millilitres of the filtrate from the tail of the funnel do not produce a precipitate or opalescence when tested with a few drops of the distillate containing nicotine (see A-3.1). Transfer the filter paper containing the precipitate to a tared platinum crucible, dry carefully and ignite the filter paper until all carbon is oxidized. Finally heat the platinum crucible over a Meker burner for not more than 10 minutes. Cool the crucible in a desiccator and weigh.

NOTE — If the nicotine content of the material is very low, an aliquot containing at least 0.01 g of nicotine should be used.

4 CALCULATION

A-4.1 Nicotine content of the material,

percent by mass =
$$\frac{11.41 \, m \, V}{M \times v}$$

where

m = mass, in g, of the residue (see **A-3.2**);

V = total volume, in ml, of the concentrated distillate after making up at room temperature (see A-3.1);

M = mass, in g, of the material taken for steam distillation (see A-3.1); and

v = volume, in ml, of the aliquot of the filtrate taken for the precipitation (see A-3.2).

ANNEX B

(*Clause* 2.3)

SEDIMENTATION TEST

B-1 PROCEDURE

Transfer 10 ml of the material to a 100 ml conical flask. Add to it 50 ml of water and mix the contents of the flask by vigorously shaking for about five minutes. Allow the flask to stand at rest for one hour and examine for any sediment at the bottom of the flask.

B-2 REPORT

The material shall be deemed to have satisfied the requirement of the test if there is no sign of any sediment at the bottom of the conical flask.

ANNEX C (Foreword)

COMMITTEE COMPOSITION

Pesticides Sectional Committee, FAD 01

Organization Representative(s) Directorate of Plant Protection Quarantine and Storage, DR RAVI PRAKASH (Chairperson) Faridabad All India Biotech Association, New Delhi SHRI SAURABH SINGHAL SHRI SHAH JI DHAR (Alternate) Central Insecticide Board and Registration Committee, Faridabad **SECRETARY** DR VANDANA SETH (Alternate) Central Insecticide Laboratory, Faridabad DR ARCHANA SINHA SHRI SUBHASH CHAUDHARY (Alternate) Consumer Guidance Society of India, Mumbai SHRI SITARAM DIXIT DR M. S. KAMATH (Alternate) Crop Care Federation of India, New Delhi DR J. C. MAJUMDAR Crop Life India, New Delhi SHRI ASITAVA SEN MS NIRUPAMA SHARMA (Alternate) CSIR -Indian Institute of Toxicology Research, Lucknow DIRECTOR DR SHEELENDRA P. SINGH Food Safety and Standards Authority of India, New Delhi ADVISOR (STANDARDS) FMC India Pvt. Limited, Bengaluru SHRI CHIRAG PATEL IDMA Laboratories Limited, Chandigarh DR INDRA RAI DIRECTOR Indian Agricultural Research Institute, New Delhi Indian Institute of Packaging, Mumbai DR TANWEER ALAM Indian Pest Control Association, New Delhi SHRI UDAYAN GHOSH Institute of Pesticide Formulation Technology, Gurgaon DR M. VAIRAMANI Ministry of Agriculture, Department of Agriculture, Chennai JOINT DIRECTOR OF AGRICULTURE (RES.) DEPUTY DIRECTOR LAB (Alternate) National Centre for Integrated Pest Management, New Delhi DR SUMITRA ARORA National Institute of Plant Health Management, Hyderabad DR MAHESH SAINI Ms T. SRIDEVI (*Alternate*) Pesticide Manufactures and Formulators Association DR ARCHANA SRIVASTAVA of India (PMFAI), Mumbai DR UDAY KUMAR (*Alternate*)

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IS 1055: 2023

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Amendments Issued Since Publication

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